

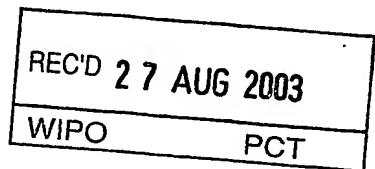
Rec'd PCT/PTO 29 DEC 2004
PCT/EP 03 / 06640



Europäisches
Patentamt

European
Patent Office

Office européen
des brevets



Bescheinigung

Certificate

Attestation

Die angehefteten Unterlagen stimmen mit der ursprünglich eingereichten Fassung der auf dem nächsten Blatt bezeichneten europäischen Patentanmeldung überein.

The attached documents are exact copies of the European patent application described on the following page, as originally filed.

Les documents fixés à cette attestation sont conformes à la version initialement déposée de la demande de brevet européen spécifiée à la page suivante.

Patentanmeldung Nr. Patent application No. Demande de brevet n°

02078294.2

**PRIORITY
DOCUMENT**
SUBMITTED OR TRANSMITTED IN
COMPLIANCE WITH RULE 17.1(a) OR (b)

Der Präsident des Europäischen Patentamts;
Im Auftrag

For the President of the European Patent Office
Le Président de l'Office européen des brevets
p.o.

R C van Dijk

BEST AVAILABLE COPY



Anmeldung Nr:
Application no.: 02078294.2
Demande no:

Anmeldetag:
Date of filing: 02.07.02
Date de dépôt:

Anmelder/Applicant(s)/Demandeur(s):

SOLVAY POLYOLEFINS EUROPE - BELGIUM
(Société Anonyme)
rue du Prince Albert, 44
1050 Bruxelles
BELGIQUE

Bezeichnung der Erfindung/Title of the invention/Titre de l'invention:
(Falls die Bezeichnung der Erfindung nicht angegeben ist, siehe Beschreibung.
If no title is shown please refer to the description.
Si aucun titre n'est indiqué se referer à la description.)

Radiation screening materials

In Anspruch genommene Priorität(en) / Priority(ies) claimed /Priorité(s)
revendiquée(s)
Staat/Tag/Aktenzeichen/State/Date/File no./Pays/Date/Numéro de dépôt:

Internationale Patentklassifikation/International Patent Classification/
Classification internationale des brevets:

C08J/

Am Anmeldetag benannte Vertragstaaten/Contracting states designated at date of
filing/Etats contractants désignées lors du dépôt:

AT BE BG CH CY CZ DE DK EE ES FI FR GB GR IE IT LI LU MC NL PT SE SK TR

02. 07. 2002

Radiation screening materials

(83)

The present invention relates to materials for the screening of solar radiation, particularly to those materials typically used for influencing plant growth in greenhouses or similar enclosures.

In order to control the growth of plants, it is known that temperature, light 5 intensity and spectral distribution of incoming light are critical factors. Furthermore, plants in a greenhouse will not grow so well if the variations in temperature between day and night are too large, as this causes them to develop resistance to the variations in temperature. Thus, for photosynthesis in 10 greenhouses photosynthetically active radiation (PAR) is required, whilst at the same time it is preferred to block near infra-red radiation in order to avoid excessive temperatures within the enclosed area. Furthermore it is desirable to scatter incoming radiation in order to prevent burning. Conversely, for outdoor mulch films the objective is to screen the photosynthetically active radiation whilst transmitting all other parts of the spectrum.

15 It is known to provide screening materials made of plastic for greenhouses so as to screen some of the incoming radiation. It is also known to provide for greenhouses glazing materials which contain an interference pigment to screen certain radiation. Hancock describes in "Plastic Culture", No. 79, 1988, pp. 4 to 14, that special additives in LDPE have a favourable effect on the microclimate 20 in greenhouses because of the generation of diffuse light and the creation of a favourably thermic effect in the greenhouses. Suitable additives are aluminosilicate in the form of kaolin, calcium carbonate, talc and kaolin clay. DE-A-2544245 discloses a polymethyl methacrylate glazing material which 25 contains an interference pigment for screening near infra-red radiation. However this pigment has the disadvantage that it does not transmit the ideal spectrum of radiation required by plants in a greenhouse.

30 EP-A-428,937 describes a grey-white coating composition for greenhouses consisting of a liquid polymeric carrier and reflecting particles suspended therein. These particles are aluminium platelets or mica platelets coated with titanium dioxide. The coating composition is used for temporary coatings in extreme weather conditions (summer). In winter, it can be removed by washing off with a water jet.

It is known to use nets made of plastic such as polyethylene as radiation screens. The nets are typically coated with a thin layer of aluminium to act as a reflector, which has been bonded to the polyethylene by sublimation or lamination prior to the polyethylene being cut into tapes and woven into nets.

5 Alternatively the aluminium can be incorporated into the body of the polyethylene, although this gives poorer reflectivity. Generally nets are considered superior to solid sheets as radiation screens for greenhouses, as they are more flexible, have a higher tear strength, and also a higher tensile strength at yield and break.

10 EP-A-659,198 discloses a composite material for use as a radiation screen in greenhouses which is a material comprising a transparent polymer and a green interference pigment consisting of a platelet shaped material coated with one or more metal oxides, and is typically of 200 μm thickness. This interference pigment only transmits the red and blue portions of visible light which can be 15 utilised by the plant. The composite material may be in the form of sheets or woven fabrics. It is also stated that for optimum performance, the pigment platelets should be aligned parallel to the surface and to one another.

We have discovered that it is possible to increase the screening efficiency of radiation screens formed by incorporating a pigment within a plastic substrate, 20 by stretching the substrate after incorporation of the pigment. Accordingly in one aspect, the present invention provides a composition for screening solar radiation, which comprises a transparent polymer having incorporated therein an interference pigment comprising a platelet shaped material, 25 wherein the polymer has been stretched in at least one direction to at least twice its original length in that direction after incorporation of the pigment coating.

Prior art sheets incorporating pigment are typically of the order of 100 μm thick. Platelets of pigment are typically of the order of 15-50 μm in length. Thus 30 the platelets can be aligned in any direction. We have found that by stretching the material, the thickness of the material can be reduced to less than 50 μm , typically less than 30 μm , thereby forcing all the pigment platelets to lie flat, parallel to one another. This significantly improves the reflective power of the pigment.

A further aspect of the invention provides a process for the production of a 35 composition for screening solar radiation which comprises a transparent polymer incorporating a interference pigment comprising a platelet shaped material,

which process comprises the steps of incorporating the interference pigment into the polymer, and then stretching the resultant polymer in at least one direction to at least twice its original length in that direction.

5 Preferably the degree of stretching of the material is to at least 4 times its original length; typically from 6 to 10 times, and most preferably 7 to 8 times.

In a further aspect the invention provides a composition for screening solar radiation, which comprises

- 10 a) a transparent polymer, and
- b) an interference pigment comprising a platelet shaped material,

10 which composition is less than 50 μm in thickness.

The transparent polymer is preferably selected from the group comprising low-density or high-density polyethylene, ethylene vinyl acetate copolymer, poly tetrafluoroethylene, ethylene tetrafluoroethylene, polyvinylidene chloride, polyvinylidene fluoride, polyvinyl chloride, polycarbonate, polymethacrylate or mixtures thereof. Other fluoropolymers may also be used.

The interference pigment can be a green interference pigment, which reflects purple and transmits green light, or a neutral pigment which provides simply a translucent film; although other types of pigment may be used.

20 The interference pigment is preferably a layered silicate, synthetic mica, glass platelets, ceramic platelets or silica platelets. Preferably the layered silicate is mica, pyrophyllite, sericite, talc or kaolin. It may optionally contain other additives such as metal oxides, eg tin oxide SnO_2 , titanium dioxide or zirconium dioxide.

25 The pigment content of the material of the invention is typically 0.1 to 30% by weight, preferably 0.5 to 10% by weight, based on the polymer.

The materials of the invention may additionally contain the usual stabilizers and processing aids employed with the particular polymer.

30 The material of the invention can be used in the form of films, sheets, spunbonded and woven fabrics and profiles. The composite material according to the present invention can be used, for example, in the form of films or plates for greenhouses, as tunnel films and mulch films. Furthermore, it can be used in the form of film for covering of soils or as substrate film.

35 It can be produced using the customary plastic processes, such as film blowing, film casting, coextrusion, cast film extrusion or lamination in the form of films, multilayer films or sheets. For the effectiveness of the pigment, it is immaterial whether the polymer is completely coloured or the pigment is present

in one or more layers of a multi layer system. Such multilayer systems can be produced by coextrusion, coating, extrusion coating, laminating or printing.

It is preferred that the material of the invention is formed into tapes or monofilaments which are then woven into nets. In a typical process the material 5 is first compounded and then blown into a film, after which it is stretched, typically to 4-8 times its original dimension. The stretched film is then cut into tapes which are woven or knitted into nets. This is known as the Lenzing process. In an alternative process (the Iso process), the blown film is cut into 10 tapes prior to stretching. Alternatively the material is extruded into monofilaments, which are then stretched prior to weaving.

Typically, the polymer utilised is fed into an extruder together with the pigment, and the mixture extruded to form a film. In an alternative embodiment, some of the polymer is compounded with a relatively high concentration (eg 30 15 wt%) of the pigment to form pellets, which are then introduced into an extruder together with additional pure polymer. Where a blown film is required, the polymer exits the extruder via a circular die, and the film is blown from a nozzle in the centre of the die. For flat films, the polymer exits the extruder via a flat die. Flat films are generally thicker than blown films.

Typically the film is then stretched at this stage. Stretching is usually 20 carried out in an oven or on a hot plate at a temperature of 100-110°C, at a stretch ratio preferably between 7 and 8 for blown films, and 4 to 6 for flat films. Optionally the stretched film may then be annealed.

If it is desired to make nets, the film is slit into tapes, which are knitted or 25 woven into a net.

The invention will now be described in more detail, with reference to Figures 1 and 2, which show plots of the degree of radiation absorption against wavelength for the films and tapes prepared in the Examples below.

EXAMPLE 1

30 70% by weight of an ethylene/butene copolymer ELTEX® (available from Solvay Polyolefins Europe-Belgium SA) was compounded together with 30% by weight of the pigment IRIODIN® SHR 9875 (available from Merck KGaA) to form pellets. IRIODIN® SHR 9875 contains pigment particles of mica, titanium dioxide, tin dioxide and zirconium dioxide 5-25µm in size, and reflects purple 35 light and transmits green light. These pellets were then incorporated with ELTEX® A5006FN1280 high density polyethylene at a weight ratio of 1:14

during extrusion, to give a final polymer containing 2 wt% of pigment, which was extruded into a film.

EXAMPLE 2

5 Example 1 was repeated, except that the final film containing 2 wt% pigment was slit into tapes and then stretched in an oven at 110°C to 7 times its original length.

EXAMPLE 3

Example 1 was repeated, except that the pigment used was IRIODIN® SHR 9870, in which the particle size is 10-60 μ m.

10 EXAMPLE 4

Example 3 was repeated, except that the final film containing 2 wt% pigment was slit into tapes and then stretched 7 times, and then slit into tapes.

EXAMPLES 5-8

15 Examples 1 to 4 were repeated, except that the level of pigment in each case was 4 wt%.

EXAMPLES 9-10

Examples 1 and 2 were repeated, except that the level of pigment in each case was 8 wt%.

20 The absorbency per unit length of the above films and tapes was then measured with a Shimadzu UV-3101PC spectrometer. The results of these measurements are shown in Figures 1 and 2, which indicate plots degree of absorption against wavelength.

The various lines shown absorption spectra for the following:

25 Figure 1

A - unstretched ELTEX® A5006FN/1280 film with no pigment

B - stretched ELTEX® A5006FN/1280 tape with no pigment

1 - Example 1 (unstretched ELTEX® A5006FN/1280 film with 2 wt% IRIODIN® SHR 9875 pigment)

30 2 - Example 2 (stretched ELTEX® A5006FN/1280 tape with 2 wt% IRIODIN® SHR 9875 pigment)

5 - Example 5 (unstretched ELTEX® A5006FN/1280 film with 4 wt% IRIODIN® SHR 9875 pigment)

35 6 - Example 6 (stretched ELTEX® A5006FN/1280 tape with 4 wt% IRIODIN® SHR 9875 pigment)

9 - Example 9 (unstretched ELTEX® A5006FN/1280 film with 8 wt% IRIODIN® SHR 9875 pigment)

10 - Example 10 (stretched ELTEX® A5006FN/1280 tape with 8 wt% IRIODIN® SHR 9875 pigment)

5 Figure 2

A - unstretched ELTEX® A5006FN/1280 film with no pigment

B - stretched ELTEX® A5006FN/1280 tape with no pigment

3 - Example 3 (unstretched ELTEX® A5006FN/1280 film with 2 wt% IRIODIN® SHR 9870 pigment)

10 4 - Example 4 (stretched ELTEX® A5006FN/1280 tape with 2 wt% IRIODIN® SHR 9870 pigment)

7 - Example 7 (unstretched ELTEX® A5006FN/1280 film with 4 wt% IRIODIN® SHR 9870 pigment)

15 8 - Example 8 (stretched ELTEX® A5006FN/1280 tape with 4 wt% IRIODIN® SHR 9870 pigment)

A comparison of Examples 1 and 2 in Figures 1 shows that the stretched tapes containing IRIODIN® SHR 9875 absorb approximately three times as much radiation as the unstretched film when the pigment concentration is 2 wt%.

At higher pigment concentrations (Examples 5 and 6 or 9 and 10) the relative

20 increase in absorption after stretching is less, but still significant. A similar effect is shown in Figure 2 for IRIODIN® SHR 9870, comparing Examples 3 and 4 or 7 and 8.

02.07.2002

(83)

CLAIMS

1 - Process for the production of a composition for screening solar radiation which comprises a transparent polymer incorporating an interference pigment comprising a platelet shaped material, which process comprises the steps of
5 incorporating the interference pigment into the polymer, and then stretching the resultant polymer in at least one direction to at least twice its original length in that direction.

2 - Process according to claim 1, wherein following stretching the polymer is converted into a woven net of tapes or monofilaments.

10 3 - Composition for screening solar radiation, which comprises a transparent polymer having incorporated therein a interference pigment comprising a platelet shaped material,
wherein the polymer has been stretched in at least one direction to at least twice its original length in that direction after incorporation of the pigment
15 coating.

4 - Process or composition according to any preceding claim, wherein the degree of stretching of the polymer is at least 4 times its original length, preferably from 6 to 10 times.

20 5 - Composition for screening solar radiation, which comprises a transparent polymer having incorporated therein a interference pigment comprising a platelet shaped material,
wherein the polymer has a thickness of less than 50 μ m.

6 - Composition according to any one of claims 3 to 5, wherein the polymer is in the form of a woven net of tapes or monofilaments.

25 7 - Process or composition according to any preceding claim, wherein the green interference pigment comprises a layered silicate, synthetic mica, glass platelets, ceramic platelets or silica platelets.

8 - Process or composition according claim 7, wherein the layered silicate is mica, pyrophyllite, sericite, talc or kaolin.

9 - Process or composition according to any preceding claim, wherein after stretching the polymer has a thickness of less than 30 μ m.

EPO - DG 1

02. 07. 2002

(83)

ABSTRACT

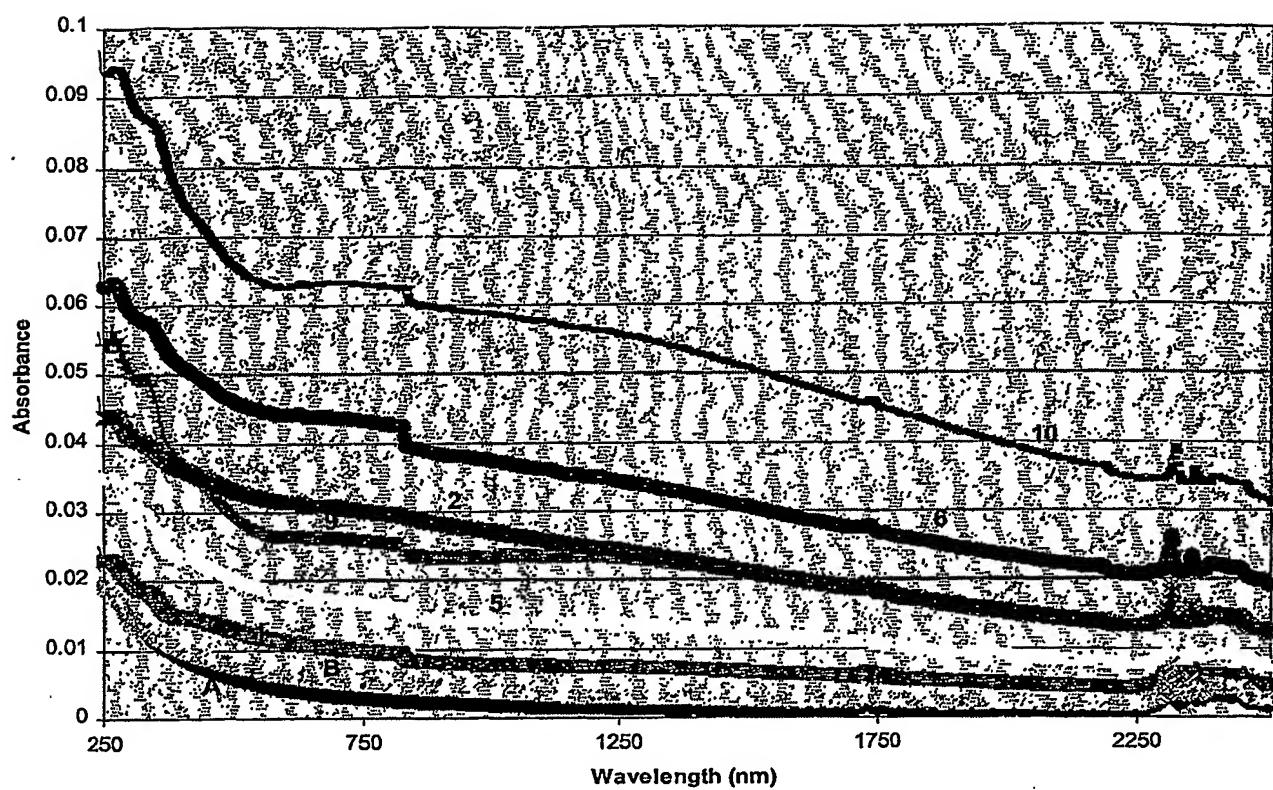
Radiation screening materials

A process for the production of a composition for screening solar radiation which comprises a transparent polymer incorporating an interference pigment comprising a platelet shaped material is disclosed, which process comprises the steps of incorporating the interference pigment into the polymer, and then stretching the resultant polymer in at least one direction to at least twice its original length in that direction. The resultant composition has improved radiation screening properties.

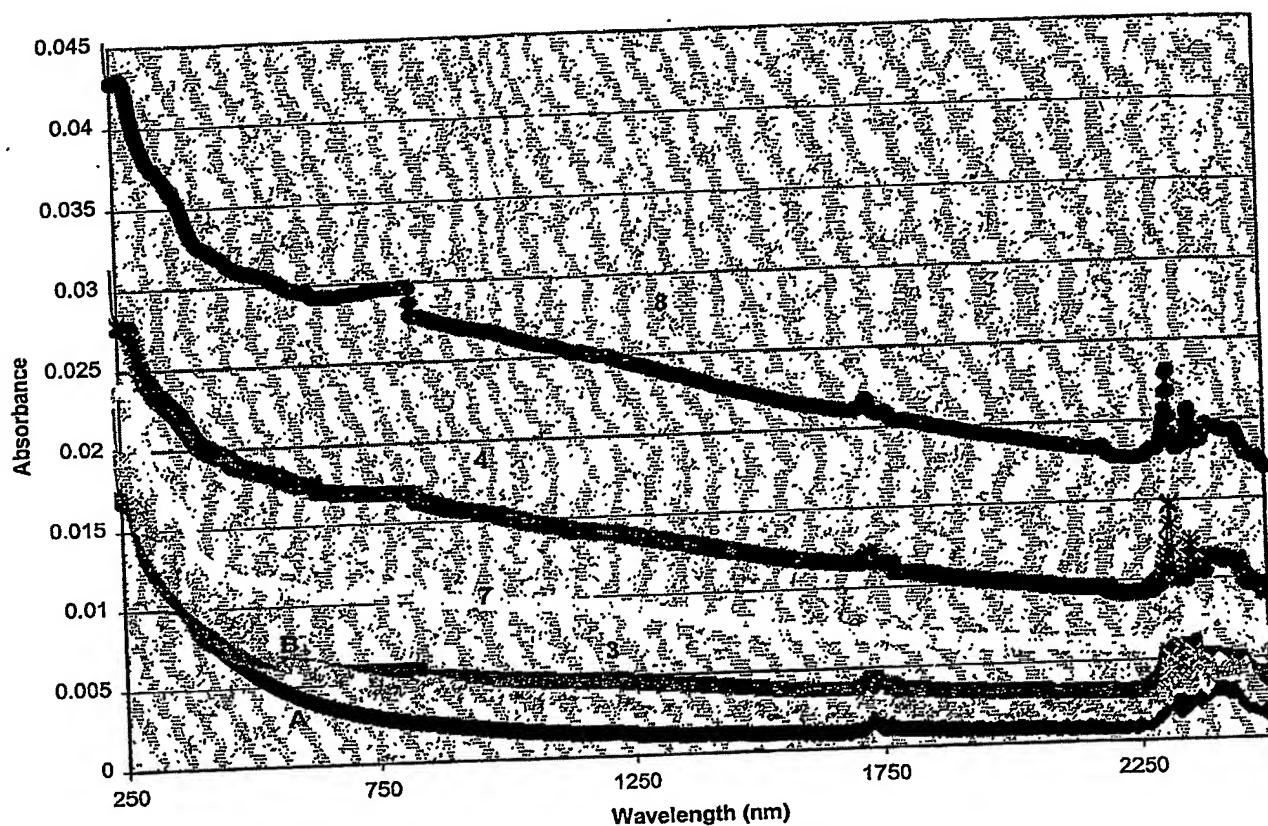
No figure.

IR - UV - Visible Spectrum

(83)



IR - UV - Visible Spectrum



**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- BLACK BORDERS**
- IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**
- FADED TEXT OR DRAWING**
- BLURRED OR ILLEGIBLE TEXT OR DRAWING**
- SKEWED/SLANTED IMAGES**
- COLOR OR BLACK AND WHITE PHOTOGRAPHS**
- GRAY SCALE DOCUMENTS**
- LINES OR MARKS ON ORIGINAL DOCUMENT**
- REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**
- OTHER:** _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.